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IS 11022 (1984): Methods of sampling and test for glues (bone, skin/fleshings and fish glues) [PCD 12: Plastics]



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*Indian Standard*

METHODS OF  
SAMPLING AND TEST FOR GLUES  
( BONE, SKIN/FLESHINGS AND FISH GLUES )

UDC 665.932/935 : 620.113



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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

# Indian Standard

## METHODS OF SAMPLING AND TEST FOR GLUES ( BONE, SKIN/FLESHINGS AND FISH GLUES )

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## IS : 11022 - 1984

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# *Indian Standard*

## METHODS OF SAMPLING AND TEST FOR GLUES ( BONE, SKIN/FLESHINGS AND FISH GLUES )

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 26 July 1984, after the draft finalized by the Adhesives Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

**0.2** The methods are intended to apply to adhesives, manufactured from materials of animal origin, such as bone, skin/fleshings and fish glues.

**0.3** The relative importance of the tests to be employed depends very largely on the purposes for which the glue is intended. In practice, it is customary for the user to determine by experience the most suitable type of glue for his purpose and then only relevant properties need to be checked. The manufacturer may also suitably advise the user keeping in view the user's needs.

**0.4** In the preparation of this standard, assistance has been obtained from BS 647 : 1981 ' Methods for sampling and testing glues ( bone, skin and fish glues ) ', published by the British Standards Institution. Assistance has also been obtained from Indian Council of Agricultural Research, New Delhi and Central Leather Research Institute, Madras in respect of apparatus used for determining Jelly Strength.

**0.5** In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960\*.

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### 1. SCOPE

**1.1** This standard prescribes the methods of sampling and test for adhesives manufactured from materials of animal origin. It applies to adhesives in the form of liquid, jelly, cake, flakes, powder or granular solids.

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\*Rules for rounding off numerical values (*revised* ).

**1.1.1** For certain uses there could be specialized tests which may be agreed between the purchaser and the supplier. Such tests have not been included in this standard.

## **2. TERMINOLOGY**

**2.1** For the purpose of this standard, definitions given in IS : 3434-1965\* shall apply.

## **3. QUALITY OF REAGENTS**

**3.1** Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1977† ) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

## **4. SAMPLING**

### **4.1 General Requirements of Sampling**

**4.1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**4.1.1** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**4.1.2** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**4.1.3** The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

**4.1.4** Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

### **4.2 Scale of Sampling**

**4.2.1** Lot — All the containers in a single consignment of the material of the same grade drawn from a single batch of processing shall constitute a lot. If a consignment is declared or known to consist of different batches of processing, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**4.2.1.1** Tests for determining the conformity of the lot to the requirements of the specifications shall be done on each lot separately.

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\*Glossary of terms for adhesives and pressure sensitive adhesive tapes ( *first revision* ).

†Specification for water for general laboratory use ( *second revision* ).



**4.2.2** The number of containers ( $n$ ) to be sampled shall be in accordance with col 1 and 2 of Table 1.

**TABLE 1 NUMBER OF COTAINERS TO BE SELECTED FOR SAMPLING**

LOT SIZE ( $N$ )	NUMBER OF CONTAINERS TO BE SELECTED ( $n$ )
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7

NOTE — When the lot size is less than 3, the number of containers to be selected shall be as agreed between the purchaser and the supplier.

**4.2.3** The containers shall be selected at random and in order to ensure the randomness of selection, a random number table (*see* IS : 4905-1968\*) shall be used. In case such a table is not available, the following procedure may be adopted:

Starting from any container, count them 1, 2, 3, ..., etc, up to  $r$  and so on in a systematic manner, where  $r$  is the integral part of  $N/n$ . Every  $r$ th container thus counted shall be withdrawn from the lot.

### 4.3 Test Samples and Referee Sample

**4.3.1** Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 1). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics and shall be not less than 200 g.

**4.3.1.1** *Sheet and cake glue* — A representative piece shall be broken from each sheet or cake. These pieces shall then be broken up smaller and reduced by mixing and quartering.

**4.3.1.2** *Pearl, cube, powdered, granulated, flake and kibbled glues* — The material shall be thoroughly mixed with the sample taken from the bulk.

\*Methods of random sampling.

**4.3.1.3 Slab and jelly glues** — A representative piece shall be cut from each slab or portion. These pieces shall be cut into smaller portions and reduced by mixing and quartering.

**4.3.1.4 Liquid glue** — The representative portions shall be bulked in a vessel, mixed by stirring, and a final sample drawn.

**4.3.1.5** Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 500 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third as the referee.

**4.3.1.6** The referee sample shall consist of the composite sample marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place for a period as agreed between the purchaser and the supplier and shall be used in case of dispute between the two.

**4.3.2 Preparation of Final Sample** — The final sample shall be ground down to a grist of approximately 3 mm by hand in an iron mortar ( or alternatively in a suitable laboratory disintegrator, the type of which shall be as agreed between the manufacturer and the purchaser ). This sample shall then be quartered, care being taken that a representative amount of all grists is included in the quartering. The powdered sample so obtained shall be kept in an air-tight container.

**4.3.2.1** In view of possible loss of moisture in grinding, it is preferable to take a separate sample for the moisture content test from the quantity obtained by the preliminary breaking up, before putting through the disintegrator. This separate sample can then be powdered by hand in a pestle and mortar, or cut with scissors, and is suitable for the moisture content test.

**4.4 Criteria for Conformity** — Criteria for conformity shall be as given in the individual specifications.

## **5. DETERMINATION OF MOISTURE CONTENT**

**5.0 General** — Small samples of powdered glue change their moisture content rapidly with change in atmospheric humidity. Even a small change in moisture content will affect the result of some of the tests, for example, an increase of 1 percent moisture would result in a decrease of about 2.5 percent in jelly strength determined on Bloom type gelometer. To avoid unnecessary dispute from this source it is recommended that when jelly strength and viscosity are reported, the result of moisture content should also be given, if an accurate comparison is required. Other properties are not affected significantly by changes in moisture content.

## 5.1 Definition

**5.1.1** For the purpose of this standard, the moisture content shall be the percentage loss in mass of the sample when a thin and evenly distributed film of glue is dried at  $105^{\circ}\text{C}$  for  $18 \pm 1$  hours to constant mass.

## 5.2 Procedure

**5.2.1** Break up the glue in an iron mortar, and pass sufficient quantity through 2·00 mm IS Sieve [ see IS : 460 ( Part I )-1978\* ]. Weigh one gram of this powder into a tared, flat bottomed stainless steel dish ( 70 mm diameter, 15 mm high and mass not less than 20 g ), preferably fitted with a cover to be used during cooling and weighing. Add 10 ml of distilled water and allow the glue to soak. Then place the dish on a water bath so that the glue is dissolved and a homogeneous solution obtained, and leave it until most of the water has evaporated. Transfer the dish to an oven maintained at a temperature of  $105\cdot0 \pm 1^{\circ}\text{C}$  and allow to remain for  $18 \pm 1$  hours. During this period the oven door shall not be opened. An electrically heated oven with thermostatic control at  $105^{\circ}\text{C}$  is suitable for this test. The thermometer bulb shall be placed 12 mm above the centre of the dish, and the  $105^{\circ}\text{C}$  mark shall be approximately 50 mm above the outside top of the oven. Remove the dish from the oven, and allow it to cool in a desiccator and weigh the dish. Replace the dish in the oven, and allow it to remain there further for 30 minutes, after which again cool and weigh; if the mass is constant to one milligram, the glue shall be considered dry.

NOTE — Every precaution shall be taken to prevent ingress of moisture to the dried film both before and during weighing.

**5.3 Calculation** — Calculate the moisture content as a percentage by mass using the equation:

$$\frac{M_0 - M_1}{M_0} \times 100$$

where

$M_0$  = the mass of original sample, and

$M_1$  = the mass of the sample after drying.

Express the result as the mean of the two values obtained in duplicate determination.

## 6. DETERMINATION OF JELLY STRENGTH

**6.0 General** — Jelly strength is generally included as a general index of the quality of the glue. Where there is no agreed sample glue for comparison, the determination of jelly strength is more difficult and it is

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\*Specification for wire cloth test sieves ( second revision ).

necessary to adhere to an arbitrary set of containers. None of the jelly strength instruments at present available is free from criticism but it is recommended that the bloom jelly strength test be regarded as the official method. This gives the mass in g necessary to produce, by means of a standard plunger of 12.7 mm diameter, a 4-mm depression in a jelly of 12.5 percent concentration by mass matured for 16 hours to 18 hours at 10°C. The directions given shall be followed implicitly.

## 6.1 Apparatus

6.1.0 The following apparatus, as shown in Fig. 1, is required.

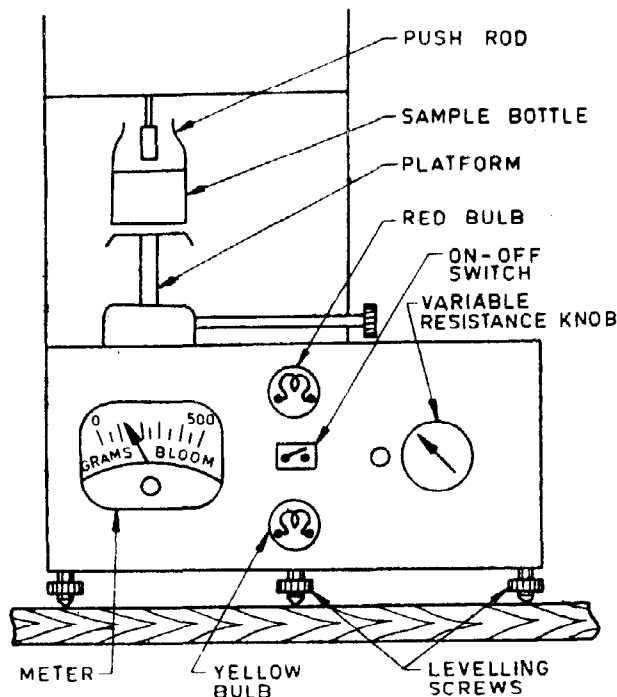


FIG. 1 GELOMETER

6.1.1 *Test Bottle* — Wide mouthed glass bottle having the following dimensions and capable of taking a rubber stopper with a diameter of 42 to 45 mm:

Capacity	155 ml
Internal diameter of the body	$59 \pm 1$ mm
External diameter of the body	66 mm
Overall height	85 mm

**6.1.1.1** Cut the tapered stopper in half and perforate the upper portion by plunging through it, at the centre, a red hot 25 mm brad. The upper half of the stopper is used to obtain a snug fit in the neck of the bottle and the air vent prevents the stopper from being blown out during the melting and heating of the sample. The test bottle and stopper shall be clean and dry.

## 6.2 Procedure

**6.2.1 Preparation of Jelly** — Weigh 15 g of the powdered sample ( *see 4.3.2* ) and transfer without loss to the sample bottle ( *see Fig. 1* ). Then add 105 ml of distilled water at approximately 15°C while thoroughly stirring the glue with a thin metal rod. Place the bottle in a bath maintained at a temperature of 10 to 15°C and allow to soak for 3 hours.

**6.2.1.1** To prevent caking and to preserve uniformity in heating and cooling, the following procedure shall be adopted both when heating the sample to 66°C and after solution when cooling prior to putting in a bath:

Place the bottle for 15 minutes in a water bath at a temperature of approximately 30°C. Bring the sample to a temperature of 60°C in the melting bath, the temperature of which shall not be allowed to exceed 70°C. Determine the temperature of the sample with an accurate thermometer placed in the glue solution and carried by a stopper having a small perforation off the centre for this purpose.

**6.2.1.2** The time required to bring the sample up to the specified temperature shall not exceed 15 minutes. After closing the bottle with the stopper carrying the thermometer and before reaching the final temperature, make the solution thoroughly uniform, preferably by swirling the bottle a number of times. Any motion that will produce violent agitation of the solution shall be avoided.

**6.2.1.3** Place the finger over the perforation in the stopper and invert the test bottle several times to mix in the water that has condensed on the walls of the bottle and the underside of the stopper. Then place the container in a constant temperature chill bath maintained at a temperature of  $10.0 \pm 0.2^\circ\text{C}$  for 16 to 18 hours. The thermostat shall be totally enclosed.

## 6.2.2 Operation of the Gelometer

**6.2.2.1** Adjust the gelometer to give a 4-mm depression and check before use.

**NOTE** — Specific directions for making this adjustment are accompanied with each instrument.

**6.2.2.2** Mount the gelometer on a rigid support and level by adjusting the three levelling screws on which the instrument rests and checking with the spirit level incorporated with the instrument.

**6.2.2.3** Turn the variable resistance knob to zero position and switch on the instrument. Standardize the instrument ( as detailed in the instruction manual provided with it ), using 150 and 350 g bloom standard module, to give these respective readings. The instrument is now ready for measurement of jelly strength of the sample.

### **6.2.3 Testing**

**6.2.3.1** To test the jelly strength of the sample, keep the variable resistance knob at zero. The yellow indicator lamp will not glow. Keep the sample well centered on the platform meant for the sample, which has been sufficiently lowered to receive the sample bottle. Gently raise the platform until the yellow indicator lamp glows. This indicates that the push rod is just in contact with the jelly. Gently turn the variable resistance knob clockwise. This will result in the yellow lamp being momentarily off and glowing when the push rod has dipped 4 mm into the jelly.

**6.2.3.2** Read the jelly strength in grams bloom directly either from the calibrated knob or from the meter depending on the design of the instrument.

**6.2.3.3** If the sample gives a jelly strength of less than 100g, prepare the jelly at a higher concentration, namely, 25 g per 100 ml of water, and make a note of this in the report.

**6.2.3.4** If the sample gives a jelly strength of above 400 g, prepare the jelly of 6.66 percent concentration, and make a note of this in the report.

## **7. DETERMINATION OF VISCOSITY**

### **7.1 U-Tube Viscometer Method**

**7.1.1 General** — The viscosity of a sample of the glue is determined at 60°C with a U-tube viscometer.

**7.1.2 Apparatus** — The following apparatus is required.

**7.1.2.1** Any type of glass capillary standard U-tube viscometer is recommended. It is essential that the time of efflux should be within the accurate range of the instrument chosen, which would normally be No. 2 viscometer. The instructions given in the specification should be adhered to. Other types of viscometers may be used, provided they are accurately standardized in SI units. The minimum time of flow for accurate work is usually 100 seconds.

**7.1.2.2** Thermostatic bath, capable of being maintained at  $60 \pm 0.2^\circ\text{C}$ .

**7.1.3 Procedure** — Place 15 g of the powdered sample ( *see* 4.3.2 ) in a corked bottle or flask and add 105 ml of cold, distilled water. Cover the bottle and allow the glue to soak until completely swollen. Then heat it on a water bath for 10 minutes maintained at  $60 \pm 0.2^\circ\text{C}$  while stirring constantly so that all the glue is completely dissolved. Pour the liquid through a funnel, loosely plugged with cotton wool into the viscometer and allow to stand in the thermostatic bath, maintained at  $60 \pm 0.2^\circ\text{C}$  for 15 minutes before taking measurement. A  $G_2$  sintered glass filter may be used instead of the cotton wool plug.

**7.1.4 Expression of Results** — Express the results as the viscosity in millipoises of the glues at a concentration of 12.5 percent by weight and a temperature of  $60^\circ\text{C}$ .

## 8. DETERMINATION OF MELTING POINT

**8.1 General** — A cup containing the glue is suspended by a glass rod held in the glue in a beaker of water which is heated slowly until the cup falls from the rod.

### 8.2 Apparatus

**8.2.1 Brass Cup** — 22 mm in height, 17 mm external diameter at the bottom. Its thickness is such that the mass is 7.0 g. Into this fix loosely a glass rod 40 mm long and 3 mm in diameter, which is flattened at one end to a disc of 9 mm diameter, and fashioned at the other end into a hook.

**8.2.2 Thermostat Bath** — Maintained at  $10.0 \pm 0.2^\circ\text{C}$  ( for example, the jelly testing chill bath ) ( *see* 6.2.1.3 ).

**8.3 Procedure** — Place 37.5 g of the powdered sample ( *see* 4.3.2 ) in a beaker and add 75 ml of cold distilled water stirring with a thin metal or glass rod. Place a watch glass on the beaker and allow to soak for 3 hours. The temperature of soaking should not be above  $22^\circ\text{C}$ . Then heat the beaker in a water bath for about 15 minutes taking care that the final temperature of the solution does not go beyond  $60^\circ\text{C}$ . This is secured by adjustment of the bath temperature which must on no account be above  $70^\circ\text{C}$ . During this heating period gently stir the solution with a rod. Take care that the glue is completely dissolved; this may be ascertained by lifting up the beaker and inspecting through the bottom. When the sample had all dissolved remove the beaker from the water bath. Pour the glue solution into the cup, insert the rod to the base of the cup and maintain it in an upright position for 16 hours in the thermostat bath for gelation to occur. Totally immerse the cup and suspend it by means of the glass rod in a beaker of water at  $15^\circ\text{C}$ , place in a water bath at  $20^\circ\text{C}$  and heat the water in the latter so that the temperature of the water in the beaker rises at the rate of approximately  $0.25^\circ\text{C}$  per minute.

**8.3.1** The temperature of the water in the beaker at which the cup falls from the rod shall be taken as the melting point of the substance.

## 9. DETERMINATION OF WATER ABSORPTION

### 9.1 Apparatus

**9.1.1** *Thermostat Bath* — maintained at  $10.0 \pm 0.2^\circ\text{C}$ .

**9.1.2** *Sieves* — 600-micron and 2-mm IS Sieves [ see IS : 460 ( Part 1 )-1978\* ].

**9.1.3** *Graduated Cylinders* — 500-ml capacity, conforming to IS : 878-1975†.

**9.2 Procedure** — Reduce the powdered sample ( see 4.3.2 ) further, and collect by means of screening the portion of the material larger than, 600-micron and smaller than 2 mm. Weigh 2.00 g of the powder so obtained and transfer it to a beaker containing 500 ml of water. Stir any particles rising to the top and allow to stand for 28 hours. Pour the contents of the beaker through a bed of moistened glass wool and allow the water to drain into 500-ml graduated cylinder. Read the volume of water collected in graduated cylinder and calculate, by deducting that volume from 500, the number of millilitres of water retained by the glue.

**9.2.1** Report the mass of water absorbed per gram of the sample.

## 10. DETERMINATION OF FOAMING TENDENCY

**10.1 General** — This test is of interest when the glue is to be applied by special machines. The results of this test will be useful in assessing the foaming tendencies of glues, but it is not possible to simulate, in the test, all the conditions that may arise during application of the glues in practice. The results of the test should, therefore, be applied with caution.

### 10.2 Apparatus

**10.2.1** *Stoppered Glass Cylinder* — 100 ml with an air space ( above the 100-ml graduation ) of  $50 \pm 2$  ml.

**10.2.2** *Water-Bath* — 250 mm diameter and 200 mm high.

**10.3 Procedure** — Disperse  $5 \pm 0.05$  g of the glue in slightly less than 50 ml of water, transfer the mixture to the glass cylinder and dilute to the 50-ml mark. Place the cylinder in the water-bath, maintained at a temperature at  $60 \pm 0.2^\circ\text{C}$ , so that the level of water in the bath is at

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\*Specification for wire cloth test sieves ( *second revision* ).

†Specification for graduated measuring cylinders ( *first revision* ).



the 70-ml graduation mark, and leave it for 30 minutes. At the end of this period remove the cylinder from the bath and shake it vigorously with a throw of about 300 mm at a rate of three shakes per second for 1 minute. Note the volume of the foam above the 50 ml mark. Loosen the stopper and then replace the cylinder in the water-bath at  $60 \pm 0.2^\circ\text{C}$  and allow it to remain until the height of the liquid in the cylinder corresponds to 45 ml. Record the time for this 45 ml meniscus to form, together with the volume of the foam above the liquid.

NOTE 1 — It is important that this determination should be carried out in a place free from draughts.

NOTE 2 — With certain glue solutions the solution/foam interface do not reach at the 45 ml mark and, therefore, the above instructions cannot be followed. These generally clear just under the 50-ml mark and the level drops 1 to 2 ml due to the further collapse of liquid foam and then rises slightly owing to the further collapse of aerial froth. In such cases, take the volume at the lowest level and report, volume of froth at that time, for example, 10 ml in 1 minute, making a note in the report that the glue solution does not clear the 45-ml mark.

**10.4 Reporting** — Report both the time in minutes and the volume of foam in millilitres as either figures by itself is not a sufficient indication of the behaviour of the glue.

## 11. DETERMINATION OF KEEPING QUALITY

**11.0 General** — The rate of growth of fungi, yeasts, and bacteria is dependent among other factors upon temperature, and upon water and preservative concentration. A dry glue is stable in storage for many years. Once a solid glue is dissolved in water or a liquid glue is diluted more water is available for growth and the concentration of preservative, if any, is decreased. Therefore, any practical test of keeping quality should preferably be made under the intended conditions of storage and use.

**11.0.1** The keeping quality test is intended simply as an index of the behaviour of the glue in practice and is not to be taken as a substitute for a more complete bacteriological examination.

**11.0.2** Evidence of microbiological activity is sought daily in a 20 percent solution of the glue, prepared under normal non-sterile conditions and maintained at  $37^\circ\text{C}$ .

**11.1 Apparatus** — Any spatulas, beakers, etc, used to transfer the adhesive and the jars used, should be clean and dry but not necessarily sterile.

**11.2 Procedure** — For a general test prepare a 20 percent solution of the glue in distilled water by the method specified in 8.3. Allow the solution to cool to room temperature (or to just above its gelation point, whichever is higher) and transfer it to a clear glass jar or conical flask. Half fill the container and cover its mouth with polythene film to prevent drying of the sample.

**11.2.1** Store the samples in an incubator maintained at a temperature of 37°C. Remove the sample daily from the incubator, and examine immediately for putrefaction. Cool to room temperature or to below the normal gel temperature, whichever is the lower, and examine for liquefaction or mould growth. Replace the sample as soon as possible in the incubator for a further day.

**11.2.2** If required the test may be extended by maintaining a similar set of samples at room temperature ( $27 \pm 2^\circ\text{C}$ ), which condition may be more favourable for growth of some organisms.

**11.2.3** Note the times when liquefaction, putrefaction, mould growth or any combination of these occur.

## 12. DETERMINATION OF pH

**12.1 Procedure** — Dissolve 1 g of the powdered sample ( see 4.3.2 ) by shaking after careful addition to a small quantity of warm, recently boiled distilled water in a stoppered volumetric flask and dilute to a final volume of 100 ml. Shake and cool to room temperature and determine pH of this solution using pH meter.

## 13. DETERMINATION OF GREASE

**13.1 General** — After saponification of the sample with alcoholic potassium hydroxide, the ether soluble material is recovered and weighed.

### 13.2 Reagents

**13.2.1 Potassium Hydroxide** — 1 N solution in ethanol.

**13.2.2 Hydrochloric Acid** — 10 percent.

**13.2.3 Diethyl Ether**

**13.2.4 Acetone** — See IS : 170-1976\*.

**13.3 Procedure** — Soak 10 g of the powdered sample ( see 4.3.2 ) in 15 ml of cold distilled water in a porcelain basin until the water is all absorbed, and the basin on a water bath until solution is complete. Add 40 ml of the potassium hydroxide solution. Evaporate the solution on a water bath and dry the residue in an oven at 105°C. Dissolve the residue in 100 ml of the hydrochloric acid solution and keep on a steam bath for 30 minutes. Transfer the solution to a separating funnel, making up the volume to 100 ml.

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\*Specification for acetone ( second revision ).

**13.3.1** When cool, add 50 ml of ether and mix thoroughly, but not too vigorously. Care must be taken not to form emulsions. Allow to stand until completely separated and run the ethereal solution into another separating funnel. Repeat this twice with further 50 ml portions of ether. Wash the ethereal solution with cold distilled water until free from acid ( 50 ml; 3 to 4 washings). Transfer to a tared flask and evaporate the ether on a steam bath. Add 3 ml of the acetone and evaporate this off for 2 minutes on the steam bath, in a gentle current of air from a bellows. When dry, cool in a desiccator and weigh.

**13.4 Calculation** — Calculate the grease as percentage by mass of the sample. Do not correct for loss of glycerol as the original fatty acid content of the grease contained in the glue may be unknown.

## 14. DETERMINATION OF ASH

**14.1 Procedure** — Weigh accurately about 3 to 5 g of the test sample in a tared silica dish. Gently heat the dish on a hot plate until the sample is well carbonized and then increase the heat until carbonization is completed. Transfer the dish with its contents to a muffle furnace and ash to constant mass at a temperature not exceeding 500°C.

NOTE — It is essential to ash at as low a temperature as possible ( *Max* 500°C ), as many glues contain chlorides which are easily volatilized.

## 14.2 Calculation

$$\text{Ash, percent by mass} = \frac{M_2 - M}{M_1 - M} \times 100$$

where

$M$  = mass in g of the crucible,

$M_1$  = mass in g of the crucible with original sample, and

$M_2$  = mass in g of the crucible with ash.

## 15. DETERMINATION OF SULPHUR DIOXIDE

**15.1 General** — This test is important when the glue is being used for coloured paper and textiles. The sulphur dioxide released by heating a solution of the glue in aqueous sulphuric acid is collected in neutralized hydrogen peroxide solution and titrated with sodium hydroxide.

## 15.2 Apparatus

**15.2.1 Distillation Apparatus** — Comprising a special round-bottomed flask of 500 ml capacity, with a steam inlet, and an outlet connected to a double surfaced condenser of the Davies pattern ( *see* Fig. 2 ). The bottom of the condenser is connected by means of a glass adaptor to a 250-ml conical flask which acts as a receiver.

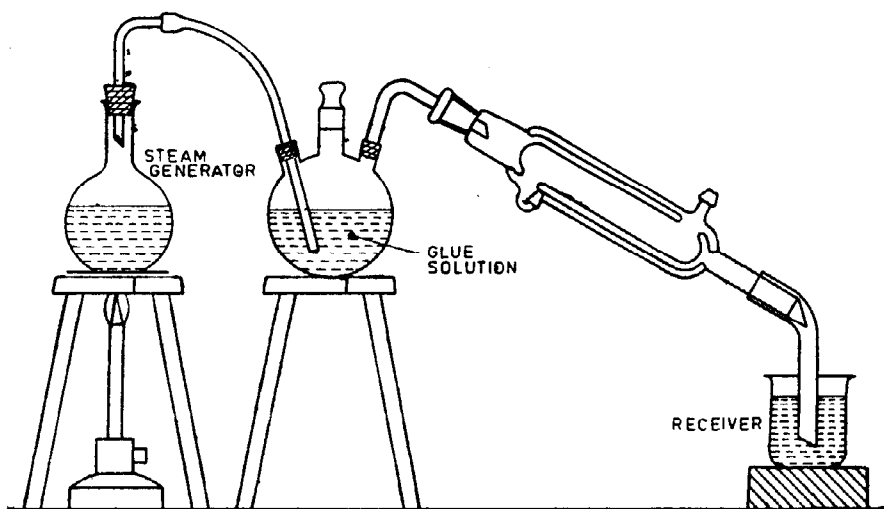


FIG. 2 APPARATUS FOR THE DETERMINATION OF SULPHUR DIOXIDE

**15.2.2 Burette** — preferably 10 ml, graduated in 0.02 ml divisions.

### 15.3 Reagents

**15.3.1 Sulphuric Acid Solution** — 20 percent ( m/m ).

**15.3.2 Hydrogen Peroxide** — equivalent to ' 10 volumes ' ( 30 g/litre ).

**15.3.3 Standard Sodium Hydroxide Solution** — 0.05 N.

**15.3.4 Screened Methyl Red Indicator** — Dissolve 0.05 g of methyl red and 0.033 g of methylene glue in 200 ml of ethanol.

**15.4 Procedure** — Place 75 ml of distilled water in the flask, introduce about 20 g of powdered glue, weighed to the nearest 0.1 g, followed by 25 ml of the sulphuric acid solution. Connect the flask to the condenser. Place 20 ml of the hydrogen peroxide solution, neutralized to the end point of the indicator together with 20 ml of distilled water, in the receiver and arrange it so that the condenser adaptor dips into the liquid. Pass a current of steam for 10 minutes, collecting about 100 ml of distillate in this time. Titrate the distillate with the sodium hydroxide solution using the recommended indicator to green end point.

**NOTE** — It is advisable to titrate in day light.

## 15.5 Calculation

$$\text{Sulphur dioxide, percent by mass} = \frac{0.16 T}{M}$$

where

$T$  = volume in ml of 0.05 N sodium hydroxide solution required, and

$M$  = mass in g of the sample taken.

## 1.6. DETERMINATION OF CHLORIDES

**16.0 General** — The chloride content is an index of the behaviour of glue under various atmospheric conditions. The calcium oxide method has been selected because it avoids loss of chlorides.

**16.0.1** To a solution of a sample of the glue calcium oxide is added, and the mixture is dried and ashed at 500°C. The residue is extracted with water and the neutralized extract titrated with silver nitrate.

### 16.1 Apparatus

**16.1.1 Basin** — made of nickel or platinum.

**16.1.2 Water Bath** — filled with boiling water.

**16.1.3 Steam Bath**

**16.1.4 Muffle Furnace** — electrically heated thermostatically controlled to give temperature range 500 to 550°C.

### 16.2 Reagents

**16.2.1 Calcium Oxide**

**16.2.2 Acetic Acid**

**16.2.3 Silver Nitrate Solution** — Dissolve 2.906 g of silver nitrate in distilled water and dilute to 100 ml.

**16.2.4 Potassium Chromate** — Dissolve 10 g of potassium chromate ( $K_2CrO_4$ ) in distilled water and dilute to 100 ml. Before use add a few drops of silver nitrate solution to every 100 ml and filter.

**16.3 Procedure** — Weigh to the nearest 0.1 g about 5 g of the powdered sample in the basin, then add 10 ml of cold distilled water and allow to soak for one hour. Heat on the water bath and add 5 g of calcium oxide. Dry on the steam bath and ignite in the muffle furnace until all organic matter is completely ashed. Extract the residue with water three times, filter and neutralize the filtrate with acetic acid. Transfer the solution, which should not amount to more

than 50 ml, to a porcelain dish and titrate it with the silver nitrate solution, using 1 ml of the potassium chromate solution as indicator.

#### 16.4 Calculation

Chlorides expressed as sodium chloride,  
percent by mass 
$$= \frac{T}{M}$$

where

$T$  = volume in ml of the silver nitrate solution required, and

$M$  = mass in g of the sample taken.

### 17. DETERMINATION OF OVERLAP JOINT STRENGTH IN LONGITUDINAL SHEAR

**17.0 General** — Joint strength in shear is an indication of whether the glue is sufficiently strong for joining purposes.

#### 17.1 Test Pieces

**17.1.1** Each test piece shall be made from the two slips of rotary cut *canarium* sp veneer, 115 mm long,  $25.0 \pm 0.3$  mm wide,  $0.318 \pm 0.15$  mm thick, joined together with the glue to be tested, so as to produce a  $25.0 \pm 0.3$  mm over-lapping joint. The veneer shall be smooth cut on at least one face. The angles of inclination of the grain of the wood across the face of any test slip shall be not steeper than 1 in 9 and the grain shall not be obviously inclined to the face that is through the thickness of the test slip. Each test slip shall be free from knots, whorls, splits, dots or other defects. The moisture content of the slips at the time of glueing shall be  $12.0 \pm 2.5$  percent, which corresponds with conditions obtaining in a normally heated room.

#### 17.2 Preparation of the Glue

**17.2.1** Weigh the powdered sample in a beaker and add the requisite amount of cold, distilled water. Place a watch glass on the beaker, and allow the glue to soak until completely swollen. The temperature of the room shall be between 15 and 22°C and the time of soaking shall be three hours. Then heat the beaker on a water bath (temperature not above 70°C) for 15 minutes, care being taken that the temperature of the solution does not exceed 60°C. During this heating period the solution shall be gently stirred with a glass rod. Care shall be taken that the glue is completely dissolved. This may be ascertained by lifting up the beaker and inspecting through the bottom. The beaker shall then be removed from the water bath.

**17.2.2** A concentration of 10 g of glue to 15 ml of water shall be used unless there is agreement between the purchaser and the supplier on other quantities, which shall be indicated in the report.

### 17.3 Preparation of Test Pieces

**17.3.1** Six test pieces shall be prepared, as detailed in **17.3.2**, **17.3.3** and **17.4.1** in two sets of three each.

**17.3.2** At the time of application of the glue, the wood shall be at ordinary air temperature, which shall not be below  $10^{\circ}\text{C}$ . The glue shall be applied with the finger, avoiding air bubbles, to one surface of each of two test slips. When the surface of the glue has become tacky or after the lapse of such time as may be specified by the manufacturer, the surface shall be placed together without rubbing, so as to produce a 25 mm overlap joint thus forming a test piece ( see Fig. 3 ).

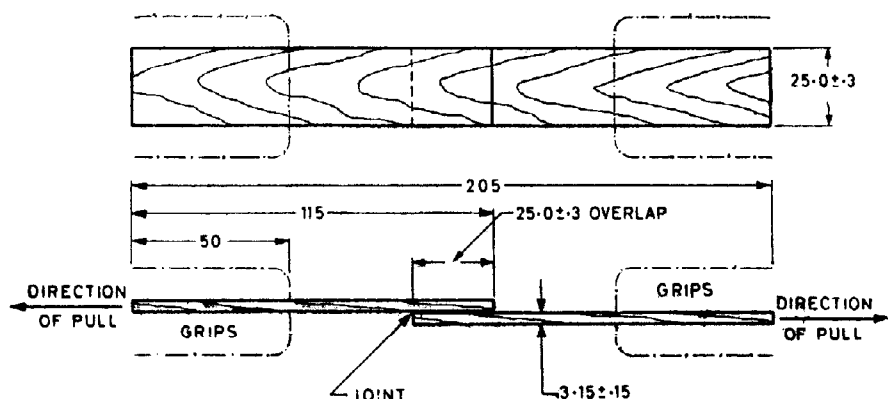


FIG. 3 TEST PIECE

**17.3.3** It is particularly important that the glued surface shall feel tacky ( almost jellied ) just below the assembling of the joint. Difficulty is sometimes experienced in hot weather, for example; if the room temperature is above  $22^{\circ}\text{C}$ , in obtaining the right degree of tackiness. In such a case the test shall not be proceeded with, as the results will be low. The time taken from the application of the glue to the closure of the joint shall not exceed 15 minutes. Each set of three joints shall be placed immediately in a clamp, a convenient form of which is shown in Fig. 4. It is important that the clamp shall apply pressure to the overlapping portion of the test slips. The pressure shall be approximately 50 kg and with the form of clamp shown in Fig. 4 may be obtained by screwing down until 'finger tight'. A convenient jig for locating the clamp and its contents is shown in Fig. 5.

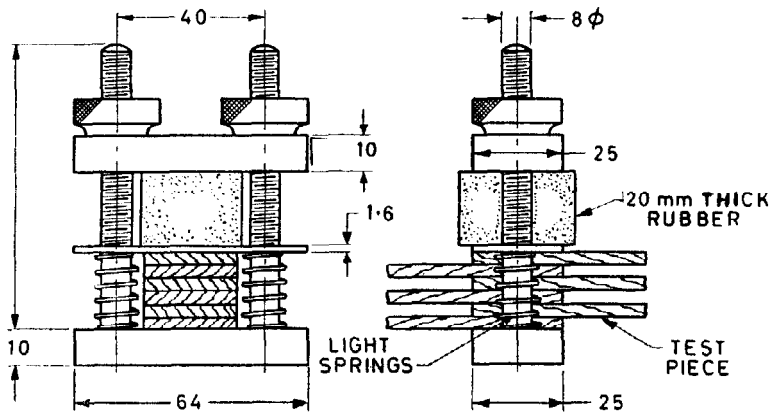


FIG. 4 CLAMPING DEVICE FOR HOLDING SIX TEST PIECES

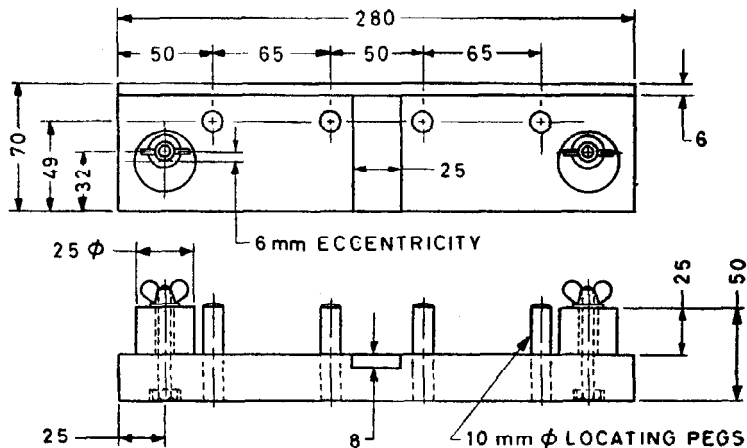


FIG. 5 WOODEN JIG FOR LOCATING TEST PIECES IN CLAMP

## 17.4 Clamping Period

**17.4.1** The joints shall remain in the clamp for 16 to 24 hours under ordinary room conditions, when they shall be removed from the clamp and freely exposed to air under the same conditions for not less than 24 hours before testing.



## 17.5 Pulling Test Pieces

**17.5.1 General** — Due to the non-axial transmission of load through the prescribed test pieces and the consequent tendency to bend during pulling, the strength which the joints develop in test is influenced by the extent to which the bending is restrained. Testing machines vary widely in this respect, one extreme being represented by the shot-loading type of machine in which the upper grips are suspended through two articulated joints from a lever free to move longitudinally, while the other extreme is typified by the standard high capacity testing machine with substantial wedge grips in a massive head in which the ends of the test pieces are maintained rigidly in line throughout the test.

**17.5.1.1** At present there is not enough knowledge of the influence of the various factors involved to enable them to be taken into account, nor can any particular testing machine be accepted as standard. Pending the development of a standard method of test, control is limited to the rate at which the load is applied to the test pieces, to the distance between the grips, and to the accuracy with which the load is measured. The rate of loading as specified under **17.5.3.2 (b)** is designed to take into account the slip experienced by wedge grips, particularly when testing joints after soaking.

**17.5.2 Accuracy of Testing Machine** — The test pieces shall be pulled in a testing machine capable of measuring the breaking load with an accuracy within  $\pm 1$  percent.

### 17.5.3 Rate of Loading

**17.5.3.1** In testing machine provided with a means of controlling the rate of increase of load, the rate of load shall be between 135 and 270 kg/per minute.

**17.5.3.2** In testing machines in which the rate of separation of the straining heads is controlled, one of the following methods shall apply:

- a) A device shall be provided incorporating a pointer moving at constant speed over a scale graduated so as to indicate a rate of increase of load within the limits of **17.5.3.1** with which the load indicator of the testing machine shall be caused to move in unison by appropriate manipulation of the rate of strain control. A scale having 45 divisions to the full circle ( corresponding to the mean rate of loading 202.5 kg/min ), Placed behind the seconds hand of a clock would be a suitable device. The necessary limits of accuracy will be maintained by keeping the pointers in agreement within  $\pm 33$  percent.

OR

- b) The straining rate shall be such that the time taken to pass from one quarter to the full specified minimum load shall be within the limits which would apply under **17.5.3.1**.

*Example* — In the case of specified minimum load of 270 kg of the time interval between  $270/4 = 67.5$  kg load and 270 kg load, that is in increasing the load by 202.5 kg shall be between  $202.5/135$  and  $202.5/270$  minutes, which is between  $1\frac{1}{2}$  and  $\frac{3}{4}$  minutes.

**17.5.4 Distance Between Grips** — The distance between the grips of the testing machines shall be between 115 to 120 mm.

**17.5.5 Computing the Failing Load** — The mean failing load for each test shall be computed from the results of a group of six test pieces.

**17.5.5.1** The mean failing load shall be stated in kg and not expressed as a stress figure of kg per sq cm.